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(71) Applicant *(for all designated States except US)*: UNIVERSITY OF LEEDS [GB/GB]; Leeds LS2 9JT (GB).

(72) Inventor; and

(75) Inventor/Applicant *(for US only)*: WILLIAMS, Richard [GB/GB]; Centre for Particle & Colloid Engineering, School of PEME, University of Leeds, Leeds LS2 9JT (GB).

(74) Agent: HARRISON, Goddard, Foote; Tower House, Merrion Way, Leeds LS2 8PA (GB).

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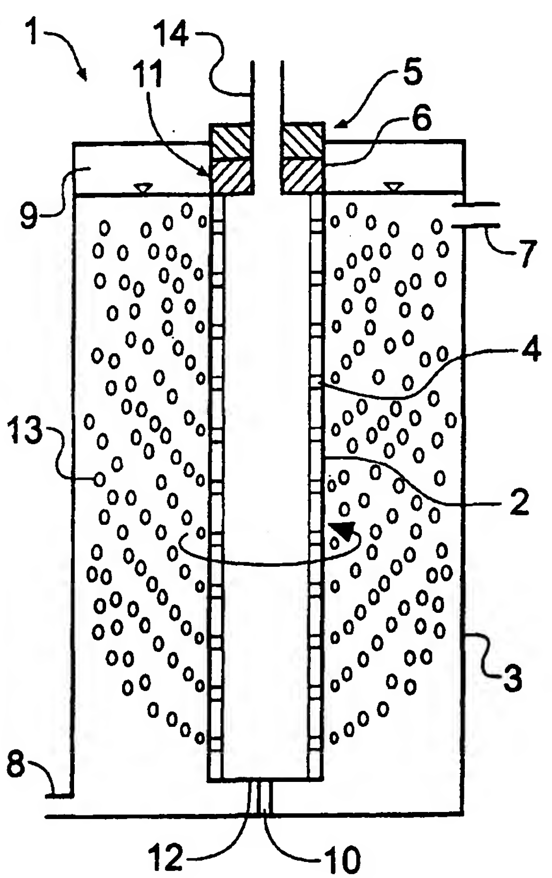
(54) Title: ROTATING MEMBRANE



(57) Abstract: An apparatus comprising a reaction vessel for accommodating a first phase and at least one membrane for accommodating a second phase, the membrane being adapted to rotate thereby creating a centrifugal force within the second phase so that the second phase is controllably dispersed into the first phase. The invention also provides for a method of controlled dispersion in the manufacture of a liquid-liquid, liquid-solid, gas-liquid, solid-semi-solid, emulsion or particulate suspension product.

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Rotating Membrane

The present invention relates to an apparatus for, and a method of, controlled dispersion of two or more immiscible phases for use in the dispersion of a liquid, gas or semisolid first phase into a gas or liquid second phase, the invention has particular use in the manufacture of liquid-liquid, liquid-solid, gas-liquid, solid-semisolid, emulsion or particulate suspension products.

Background to the Invention

Emulsion manufacturing is an important process in the food, cosmetic, chemical, pharmaceutical and mineral processing industries. There are several different conventional methods for producing dispersions of two or more immiscible phases. For example, the method can be based on agitation of the mixture, high pressure homogenisation or cross flow membranes.

The prior art method based on agitation involves vigorous stirring which establishes a turbulent flow or eddy in the mixture in the manufacturing vessel. In such systems, one phase is broken up into droplets (the discontinuous phase) and becomes suspended in the other (the continuous phase). The rotor-stator system employs tooth-disc high-speed homogenisers or colloid mills, these devices generate a high shear between a rotor and a stationary smooth, roughened or grooved surface. Turbulence is the primary cause of fluid disruption and in turn leads to droplet formation.

High-pressure homogenisation involves passage of the emulsion mixture through a narrow orifice or inject dispersion, in which two jets of different components are made to collide head-on. These processes may be assisted with the use of power, ultrasound or electrical fields. Pressures in the region of 5.0×10^6-3.5×10^7 Pa are common. In these systems separation is caused by turbulence and cavitation effects.

Cross membrane emulsification involves injection of one phase through a porous substrate (membrane) in such a way that droplets formed at the end of the pores at the membrane surface come into contact with the second (continuous) phase. Pressure can be applied across the membrane and the velocity of the cross flow can be controlled.

A problem associated with the methods based on high shear and high-pressure homogenisation is that the mean droplet size and size distribution of the droplets cannot easily be controlled. Droplet size distribution is often so wide that emulsion characteristics and stability can be effected from batch to batch and even within the same batch. This sort of variation in product quality is especially problematic for the pharmaceutical industry where exact amounts of active ingredients in medicaments is essential for patient safety. Dispersion by cross flow membranes can produce droplets of more uniform size and narrower size distribution than the other two previously mentioned methods. However, droplets emerging from the membrane have a tendency to coalesce and so particle size and size distribution will be varied to some extent. Additionally, droplets can adhere and/or spread over the membrane surface and thus clog the membrane pores especially if the discontinuous phase is viscous. Eventually, if the clogging persists not only does the overall performance of the membrane deteriorate but the droplet size and size distribution will also vary.

In the production of semi-solid droplets or particulates the use of a batch and continuous manufacturing operation can cause damage to the products due to the effects of fluid shear in circulating the continuous phase (in pipes and pumps). Circulation and transport of the products is therefore also problematic.

Another problem associated with the prior art methods is their poor reproducibility of equipment which means that product quality can vary from one manufacturing vessel to another. This problem is exacerbated when scaling up from the laboratory to small scale to large scale production. This in itself can lead to inflexibility of

manufacturing apparatus and under utilisation of expensive equipment with the consequential costs borne by the consumer.

A yet further problem with these methods is that energy utilisation for large scale production is poor and increases as the manufacturing vessel size increases so adding significantly to manufacturing costs.

An apparatus that could effectively control droplet size and size distribution of the discontinuous phase in an emulsion, whilst improving reproducibility of equipment, quality control of the product, improvement of energy utilisation and facilitation of scale-up production would offer immediate advantage over the prior art.

The present invention has overcome many of the problems associated with the prior art and provides an elegant solution by using a compartmentalised system in which the discontinuous and continuous phases are separated by a membrane, and in use the membrane spins so that the phase within the membrane compartment passes through the membrane and is subjected to a centrifugal force.

Statement of the Invention

According to a first aspect of the invention there is provided an apparatus comprising a reaction vessel for accommodating a first phase and housed therein there is provided at least one membrane for accommodating a second phase, the membrane being adapted to rotate and upon rotation the second phase is controllably dispersed into the first phase.

Preferably, the first phase is continuous and the second phase is discontinuous, alternatively the first phase is discontinuous and the second phase is continuous. The continuous phase may comprise a fluid such as a gas, liquid or emulsion and the discontinuous phase may comprise a fluid such as a gas, liquid or emulsion. The discontinuous phase, on contacting the continuous phase, may turn semi-solid or particulate by polymerisation, precipitation or some other process.

Reference herein to controllably dispersed is intended include the control of droplet size and droplet size distribution within the product.

Preferably, the reaction vessel is substantially circular in cross section and comprises a vat or pipe.

Preferably, the membrane is elongate and substantially tubular in shape.

Preferably, the apparatus comprises a plurality of membranes arranged within the reaction vessel and more preferably the membranes are evenly spaced apart. In this way the apparatus can be readily scaled up for large scale production and can maintain reproducibility of equipment.

Preferably, at least one end of the membrane is attached to an inner surface of the reaction vessel and is held in position by a membrane support means. Alternatively, the membrane can be attached at both ends to an inner surface of the reaction vessel. It will be understood that the membrane is free to rotate within the reaction vessel.

Preferably, the membrane is positioned so as to be coaxial with respect to the reaction vessel.

Preferably, the membrane is positioned within the reaction vessel in a substantially central position with respect to the reaction vessel's inner wall. The membrane may be positioned perpendicularly within the reaction vessel or in the instance of the reaction vessel comprising a pipe the membrane is positioned horizontally along the length of the pipe.

In one embodiment of the invention the membrane comprises a double concentric membrane, arranged so that a first or inner membrane is surrounded by a second membrane of suitably larger diameter so that an outer surface of the inner membrane is a selected distance from an inner surface of the second membrane.

Preferably, the membrane is formed from a ceramic material, glass, polymer based film or sintered metal such as stainless steel or any other material in which pores can be made, for example by laser drilling. In the instance of the membrane being constructed of stainless steel or the like it will preferably have a rolled surface finish and in the instance of the membrane being constructed of ceramic material it will preferably be a precision sintered ceramic. The selection of material depends on the hydrophobicity of the two phases and is not intended to limit the scope of the application.

Preferably, the membrane is provided with a plurality of pores passing radially through the material of the tube. The diameter of the pores and their distribution over the membrane surface will be determined by the type of dispersion that is desired.

Preferably, the membrane is provided with at least one inlet port to a space defined in the membrane and into which the second phase is fed. In one embodiment of the invention the membrane is a dead-end tube, in an alternative embodiment the membrane is also provided with an exit or outlet port and is in the form of a flow-through tube.

Preferably, the apparatus is provided with a power supply means or rotary drive for driving the rotation of the membrane. Additionally, the apparatus will be provided with a controller means for controlling the rotational speed of the membrane more preferably the means is a variable speed motor.

Preferably, the membrane rotates in use at a speed of 200-10,000 rpm and more preferably at a speed of 1,000-5,000 rpm.

Preferably, the apparatus is provided with pressure supply means for providing a source of pressure so as to force the second phase through the membrane.

Preferably, the apparatus is provided with means for providing circulation of the first and/or second phases. Such a means can be a stirrer.

Preferably, the apparatus is provided with means, for example a temperature control, for controlling the temperature in the first and/or second phases and may optionally be provided with a mechanical vibration unit, the unit being preferably mounted on the membrane.

According to a second aspect of the invention there is provided use of an apparatus as hereinbefore described for the production of a liquid-liquid, liquid-solid, gas-liquid, solid-semi-solid, emulsion or particulate suspension product.

According to a third aspect of the invention there is provided a method of controlled dispersion in the manufacture of a liquid-liquid, liquid-solid, gas-liquid, solid-semi-solid, emulsion or particulate suspension product comprising introducing a first phase into a reaction vessel and introducing a second phase into a membrane housed within the reaction vessel and rotating the membrane within the reaction vessel so as to create a centrifugal force within the second phase.

Preferably, a constant pressure drop is applied to the second phase so as to facilitate passage through the membrane.

Preferably, the second phase is circulated through/across the membrane until the desired concentration of discontinuous phase in the continuous phase is achieved.

Preferably, the method further includes any of the features hereinbefore described.
It will be appreciated that the membrane is located within the reaction vessel and that the phase to be dispersed flows within the membrane at a controlled rate. As droplets are formed on the surface of the membrane they may be thrown away from the spinning membrane tube and in this way productivity of the apparatus is increased. The enhanced disengagement process is based upon a density difference between the

first phase and the product formed by the second phase. Moreover, the opportunity for coalescense of droplets emerging simultaneously from adjacent pores on the membrane is reduced. The apparatus and method of the present invention is particularly suited to controlled dispersion of dense viscous fluids since the enhanced force created by rotation acts to increase the breakage of the neck connecting the droplet to the fluid reservoir behind the membrane.

The apparatus and method of the present invention is equally well suited to the production of solids and capsules since the materials that might otherwise adhere to a stationary membrane can be removed from the surface thus reducing the likelihood of droplet adhesion.

The apparatus and method of the present invention may be adapted to produce either a single-phase emulsion or an emulsion containing a plurality of discontinuous phases, moreover it may be used in either batch-process or in a continuous production mode.

According to a fourth aspect of the invention there is provided a product comprising a viscous high concentration emulsion or a polymer suspension produced by the process hereinbefore described and optionally further including any of the preferred features hereinbefore described.

Brief Description of the Drawings

The invention will now be described by way of example only with reference to the following Figures wherein:

Figure 1 represents an apparatus according to the present invention;

Figure 2 represents a first embodiment of the apparatus of the invention;

Figure 3 represents a detailed view of a membrane depicted in Figure 2;

Figure 4 represents a second embodiment of the apparatus of the invention; and

Figure 5 represents a third embodiment of the apparatus of the invention.

Detailed Description of the Invention

With regard to Figure 1 there is shown an apparatus 1 according to the present invention in which membrane 2 is housed perpendicularly within reaction vessel 3. Membrane 2 is connected at end 6 to the lid 9 of reaction vessel 3 and, if required, at end 12 to the floor 10 of the reaction vessel. The membrane is free to rotate within the reaction vessel. The membrane may be constructed of a precision ceramic, a polymer based film or a drilled membrane such as stainless steel. The selection of material depends on the hydrophobicity of the discontinuous and continuous phases. The membrane is provided with a plurality of pores 4, and in the embodiment depicted in Figure 1, it is shown to be a dead-end tube. However it will be appreciated that the tube may also be a flow through as shown in Figure 5.

In use, and once the appropriate phases have been introduced into the reaction vessel and membrane compartments, the membrane is rotated. Rotation of the membrane is driven by a variable speed motor 5 which is also provided with means to ensure a constant pressure drop down the membrane tube so that the phase housed within the membrane can be ejected through the pores at a predetermined velocity. It may optionally be provided with a mechanical vibration unit 11 that seeks to perturb the liquid ejection process so as to meter-out the dispersed phase. The apparatus can be provided with means (not shown) for controlling the temperature on either side of the membrane.

In the embodiment of Figure 1, reaction vessel 3 contains the continuous phase in addition to any other necessary chemicals/ingredients. The continuous phase is fed into the vessel or chamber via inlet port 8 and is eventually retrieved via the product

outlet port 7. Membrane 2 is fed with a discontinuous phase via inlet port 14 and when sufficient centrifugal force has been generated within the discontinuous phase within the membrane and an internal pressure is applied to the membrane, droplets 13 are forced through the membrane and thrown off into the continuous phase. In the case of an emulsion, the droplets are rapidly thrown free of the membrane surface thus reducing the potential for coalescence of emerging adjacent droplets and reducing the potential for pore clogging. In this way a controlled and improved quality of dispersion is achieved. The final product can be bled off via port 7 on reaching the desired concentration of discontinuous phase in the emulsion. If the density of the product phase is high, the material may settle downwards under gravity, in which case the inlet 8 and outlet port 7 may be transposed. A significant feature of the invention is that control over the volume concentration of the discontinuous phase product is readily adjusted by varying the ratios of the two fluid flow rates, for the continuous phase at inlet port 8 and for the discontinuous phase at inlet port 14 in addition to the withdrawal rate at outlet 7.

An example of the specific application of the technology using apparatus 1 is in the dispersion of water into oil. An enhanced efflux rate can be attained using a stainless steel membrane being spun at different speeds. The flux increases significantly with rotational speed whilst operating at a given pressure drop.

In the instance of manufacturing a solid particle, for example, by direct chemical precipitation or by a polymerisation process, the effects of the spinning membrane improves the control and specification of the size distribution and increases the production rate. Use of the equipment offers floor space savings since the manufacturing process is intensive.

It will be appreciated that the configuration of the membrane and the number of membranes provided within the reaction vessel may be varied, and is not intended to limit the scope of the application.

In another embodiment of the invention and with regard to Figures 2 and 3, there is shown a membrane 2 composed of a pair of concentric tubes 15 and 16 spaced a distance Z apart. The distance can be varied according to a user's requirements but is of a sufficient amount to allow free rotation of membrane tube 16 within tube membrane 15. The outer tube 15 is positioned within the reaction vessel and attached thereto at end 12 to the floor of the vessel 10, it is attached at an opposite end 6 to the lid of the reaction vessel 9. It is also provided with an inlet feed port 14. The membrane tube is free to rotate within the vessel in direction X.

The second or inner tube membrane 16, is positioned at end 18 to the floor of tube 15 at position 17. It is attached at an opposite end 20 and sealed from the interior of tube 15 with a seal means 21, additionally it is provided with a separate and dedicated inlet feed port 19. Tube membrane 16 is free to rotate in direction Y inside tube membrane 15. In the arrangement depicted in Figures 2 and 3 it will be appreciated that different membrane configurations are possible and equally well suited to the apparatus and method of the invention. Membranes can be composed of a series of concentric tubes i.e. more than two not shown but as generally depicted in the Figures so as to allow for multiple dispersions for example and without limitation for oil/water/oil systems.

The apparatus of the invention can also be used for dosing/dispersion reagents into product lines. For example, and with reference to Figure 4 there is shown a membrane 2 positioned in a substantially central position within pipe/product line 24. The membrane is held in position at end 6 by a positioning means 22 and inlet port 14 and is held in position at an opposite end 12 by a second positioning means 23. In this arrangement a number of membranes can be positioned along the length of a pipe/product line 24 and be made to rotate. An example would be the dispersion of a polymeric flocculant into a process stream

In a yet further embodiment of the invention, as shown with a flow through membrane in Figure 5, dispersion can be from the phase outside of the membrane 25

i.e from the reaction vessel into the inner region 26 of the membrane through which a circulating cross flow is generated.

It will be appreciated from the embodiments of the invention hereinbefore described that the rotating membrane can be a closed end design or flow through and that there may be a plurality provided within a reaction vessel or pipe/product line and that the phase to be dispersed may be housed either within or outside of the membrane.

The present invention provides significant advantage over the prior art in providing an apparatus for, and method of, improved product quality and controlled dispersion of one phase into another and is of particular use to the food, cosmetic, chemical, pharmaceutical and mineral processing industries. Moreover, the present invention allows for the generation of new products with properties that could not be achieved using conventional methods. For example, in the production of viscous high concentration emulsions and in the production of polymer suspensions.

Claims

1. An apparatus comprising a reaction vessel for accommodating a first phase and housed therein there is provided at least one membrane for accommodating a second phase, the membrane adapted to rotate and upon rotation the second phase being controllably dispersed into the first phase.

2. An apparatus according to claim 1 wherein the first phase is continuous and the second phase is discontinuous.

3. An apparatus according to claim 1 wherein the first phase is discontinuous and the second phase is continuous.

4. An apparatus according to any preceding claim wherein the continuous and/or discontinuous phase comprises a fluid or gas or liquid or emulsion.

5. An apparatus according to any preceding claim wherein the reaction vessel is substantially circular in cross section and comprises a vat or pipe.

6. An apparatus according to claim 5 wherein the membrane is positioned so as to be coaxial with respect to the reaction vessel.

7. An apparatus according to any preceding claim wherein the membrane comprises an elongate tube.

8. An apparatus according to claim 7 wherein the membrane is provided with a plurality of pores passing radially through the material of the membrane.

9. An apparatus according to either claim 7 or 8 wherein the membrane is provided with at least one inlet port to a space defined in the membrane and into which the second phase is fed.

10. An apparatus according to any one of claims 7-9 wherein the membrane is a dead-end tube, or alternatively the membrane is also provided with an exit or outlet port and is in the form of a flow-through tube.

11. An apparatus according to any one of claims 7-10 wherein the membrane comprises a double concentric membrane, arranged so that a first or inner membrane is surrounded by a second membrane of suitably larger diameter so that an outer surface of the inner membrane is a selected distance from an inner surface of the second membrane.

12. An apparatus according to any preceding claim comprising a plurality of membranes.

13. An apparatus according to claim 12 wherein the membranes are arranged within the reaction vessel so as to be evenly spaced apart.

14. An apparatus according to any preceding claim wherein at least one end of the membrane is attached to an inner surface of the reaction vessel.

15. An apparatus according to any preceding claim wherein the membrane is attached at both ends to an inner surface of the reaction vessel.

16. An apparatus according to any preceding claim wherein the membrane is positioned within the reaction vessel in a substantially central position with respect to the reaction vessel's inner wall.

17. An apparatus according to any preceding claim wherein the membrane is formed from a ceramic material, glass, polymer based film or sintered metal, for example stainless steel.

18. An apparatus according to claim 17 wherein the stainless steel membrane comprises a rolled surface finish.

19. An apparatus according to claim 17 wherein the ceramic membrane is composed of a precision sintered ceramic.

20. An apparatus according to any preceding claim further comprising a rotary drive for driving the rotation of the membrane.

21. An apparatus according to any preceding claim further comprising a controller for controlling the rotational speed of the membrane.

22. An apparatus according to either claim 20 wherein the drive is a variable speed motor.

23. An apparatus according to any preceding claim wherein the membrane, in use, rotates at a speed of 200-10,000 rpm.

24. An apparatus according to claim 23 wherein the membrane, in use, rotates at a speed of 1,000-5,000 rpm.

25. An apparatus according to any preceding claim further comprising a pressure supply for providing a source of pressure so as to force the second phase through the membrane.

26. An apparatus according to any preceding claim further comprising a means for providing circulation of the first and/or second phases.

27. An apparatus according to any preceding claim further comprising a temperature control for controlling temperature in the first and/or second phases.

28. An apparatus according to any preceding claim further comprising a mechanical vibration unit mounted on the membrane.

29. Use of an apparatus according to any preceding claim for the production of a liquid-liquid, liquid-solid, gas-liquid, solid-semi-solid, emulsion or particulate suspension product.

30. A method of controlled dispersion in the manufacture of a liquid-liquid, liquid-solid, gas-liquid, solid-semi-solid, emulsion or particulate suspension product comprising introducing a first phase into a reaction vessel and introducing a second phase into a space within a membrane housed within the reaction vessel and rotating the membrane within the reaction vessel so as to create a centrifugal force within the second phase.

31. A method according to claim 30 wherein a constant pressure drop is applied to the second phase so as to facilitate passage through the membrane.

32. A method according to either claim 30 or 31 wherein the second phase is circulated across the membrane until a desired concentration of discontinuous phase in the continuous phase is achieved.

33. A product comprising a viscous high concentration emulsion or a polymer suspension produced by the method according to any one of claims 30-32.

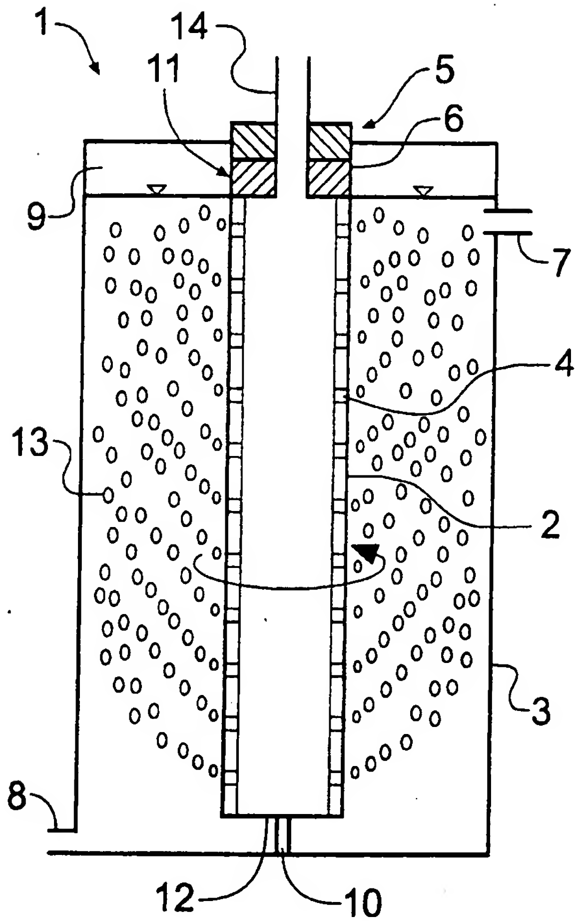

Fig. 1

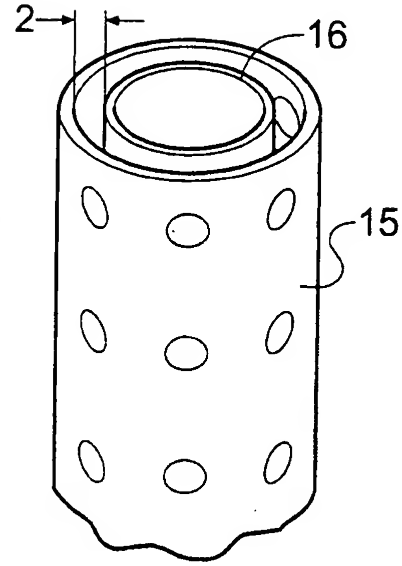

Fig. 3

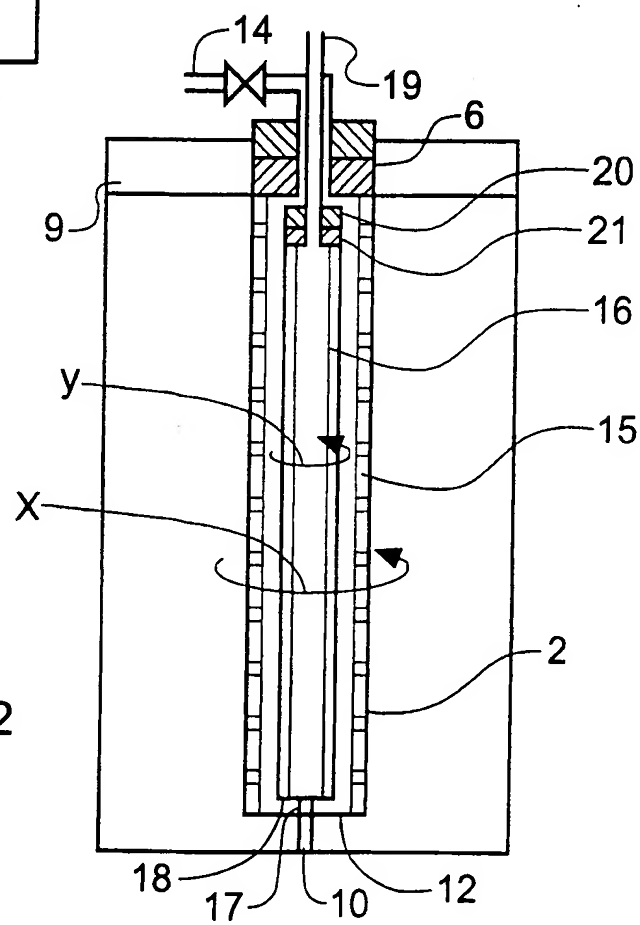

Fig. 2

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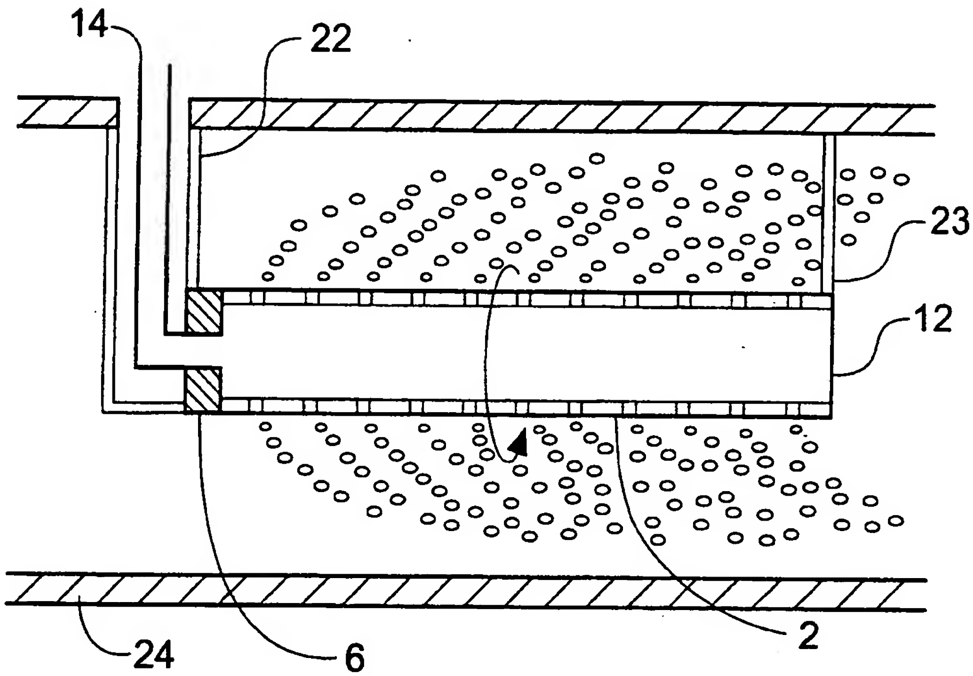

Fig. 4

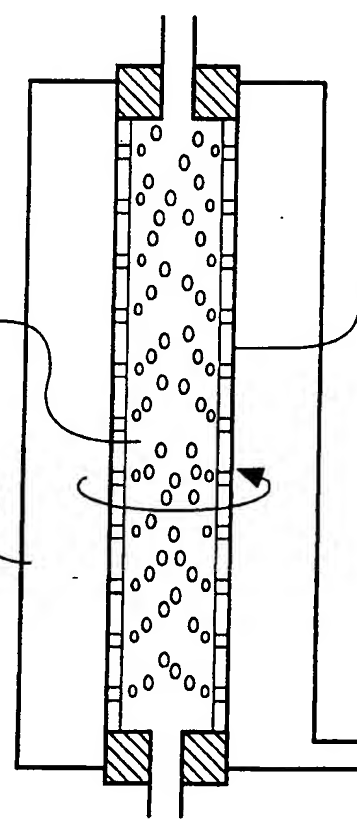

Fig. 5

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INTERNATIONAL SEARCH REPORT

Interr. nal Application No
PCT/GB 0 917

A. CLASSIFICATION OF SUBJECT MATTER
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According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01F B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US 5 874 006 A (R.LEE ET AL) 23 February 1999 (1999-02-23) abstract ---	17,18
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
9 April 2001	18/04/2001

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 0[illegible]4917

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Infor[redacted]on patent family members

Interr. nal Ap[redacted]tion No
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